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(56) Documents Cited
US 5282898 A US 5261956 A US 5232881 A
US 5129553 A US 5074475 A US 5028268 A

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(54) **Pozzolanic material for mortars and concrete**

(57) A pozzolanic material for incorporation in a concrete composition comprises a mixture of metakaolin and from 2% to 50% by weight of an aqueous medium, based on the total weight of dry metakaolin and the aqueous medium. The aqueous medium may be water or an aqueous solution containing as the solute, for example, an alkali, a dispersing agent or an agent which either accelerates or retards the setting of the concrete. The pozzolanic material is found to exhibit enhanced reactivity with calcium hydroxide as compared with conventional metakaolin.

notes on leading properties. abilities to be assessed

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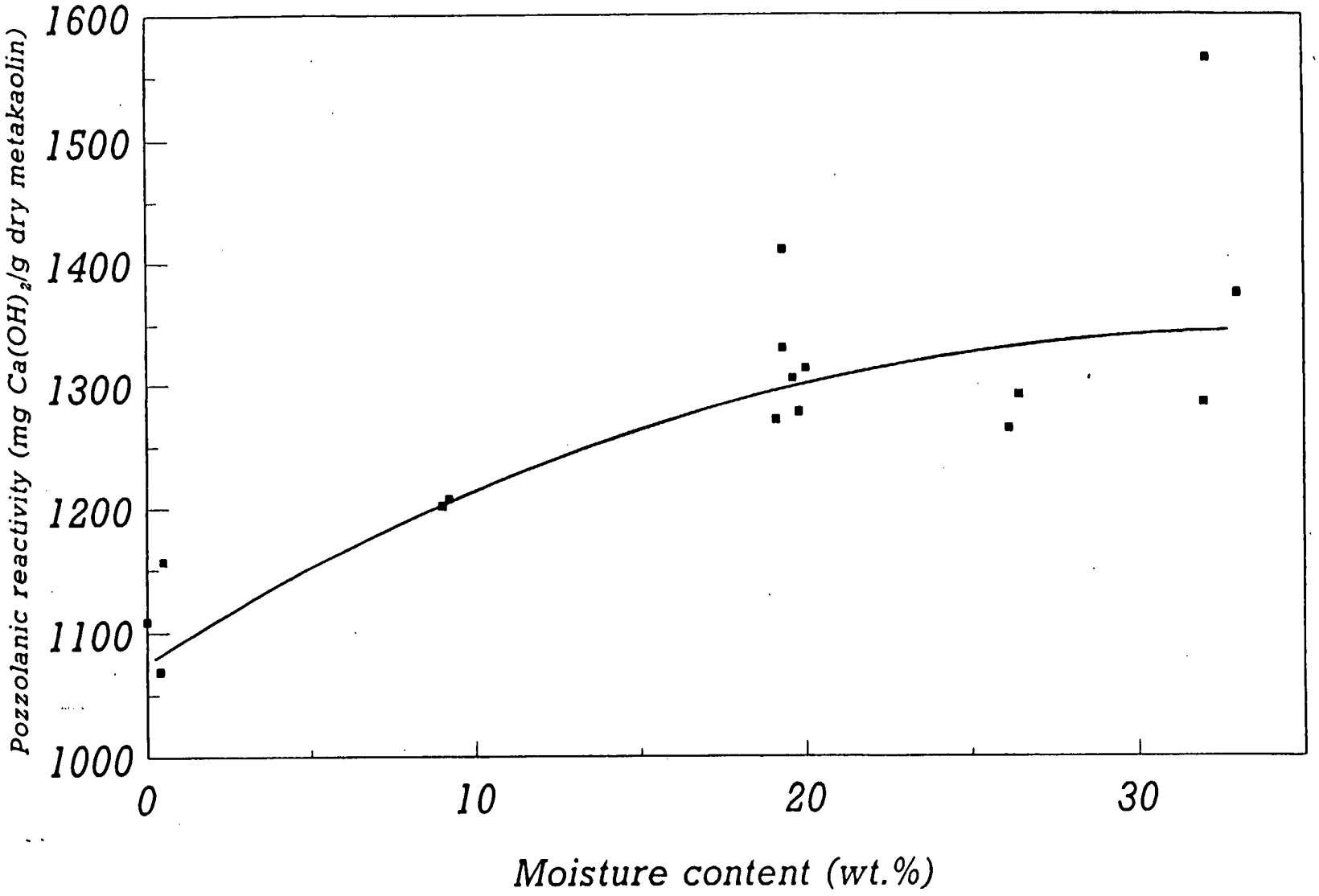
Effect of added water on pozzolanic reactivity of metakaolin



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IMPROVED POZZOLANIC MATERIAL FOR MORTARS AND CONCRETE

This invention concerns a process for improving the performance of metakaolin as a pozzolanic material in mortars and concrete.

A pozzolanic material is defined as a pulverulent siliceous or aluminosiliceous material which will react with slaked lime, or calcium hydroxide, at ambient temperature and in the presence of moisture to form a cementitious compound. An example of a natural product which has pozzolanic properties is volcanic ash, but it is now more common to use pozzolanic materials which are by-products of industrial processes such as ground granulated blast furnace slag, pulverised fly ash and condensed silica fume.

Metakaolin, the product of calcining kaolinitic clay at a temperature in the range of from 550°C to 925°C for from a few minutes to 8 hours, is one of the best pozzolanic materials presently known. It reacts very rapidly, i.e. within 14-28 days with approximately its own weight of calcium hydroxide. The present invention relates to improving still further the performance of metakaolin as a pozzolanic material.

Concrete is generally not durable when exposed to aggressive conditions. Examples of commonly occurring aggressive conditions are those which obtain where there is contact with aqueous media of low pH, for example farmyard effluents, with salt solutions, as is the case with marine structures and road bridges, or with running water. One of the main reasons for low durability is the presence of crystals of calcium hydroxide in the cured concrete. Calcium hydroxide is formed as a hydration product of Portland cement according to the formula:-

C_3S, C_2S	+	H_2O	=	CSH	+	CH
Portland cement				hydrated cement		calcium hydroxide

Unfortunately, calcium hydroxide is soluble in water with the result that it can be leached out of concrete leaving a weakened porous structure. Also it is chemically reactive and highly alkaline. It reacts with sulphate and chloride ions and can dissolve silica and glass such as is present in glass fibre reinforced concrete. In many cases the structure is weakened, and the chemical reactions lead to swelling and cracking of the concrete.

Pozzolanic materials can improve the durability of concrete by chemically combining with some of the calcium hydroxide. Stable cementitious compounds are formed which are less chemically reactive and less soluble. Typical pozzolanic materials include ground granulated blast furnace slag, pulverised fly ash and silica fume. However, these are all by-product materials and suffer from certain disadvantages, namely that they are generally inconsistent in quality and effectiveness, they have a relatively low pozzolanic reactivity, they react only slowly with calcium hydroxide, and they may contain deleterious impurities such as carbon, sulphur compounds and alkali metal ions.

The major advantages of metakaolin over other pozzolanic materials are that it can be made in a substantially pure chemical form and has no deleterious side effects, it can be manufactured in such a way that its properties and performance are consistent from batch to batch, it reacts rapidly with calcium hydroxide, and, on a weight for weight basis, it has a relatively high capacity for fixing calcium hydroxide.

It has been known for many years that metakaolin is a superior pozzolanic material. For example, a comprehensive review of Portland-pozzolan cement by G.Malquori, Fourth Intl. Symp. on the Chemistry of Cement, Paper VII-3, Washington, D.C., 1960, states that kaolin calcined at between 600 and 700°C will combine with its own weight of calcium hydroxide.

It is also known from EP-A-0333584 that metakaolin enhances the durability of glass-fibre-reinforced concrete, as a result of the metakaolin fixing the calcium hydroxide, thereby preventing the latter from reacting with, and weakening, the glass fibres.

An object of this invention is to improve still further the reactivity of metakaolin with calcium hydroxide. If this improvement could be achieved the metakaolin would react more quickly, giving a more rapid development of strength in the concrete in which it was used. It would be possible to use less metakaolin in the concrete with resultant significant cost savings. The rheological properties of the uncured concrete mix would be improved, and, in the cured concrete, strength would be enhanced and porosity and permeability would be decreased.

According to the present invention, there is provided a pozzolanic material for incorporation in a concrete composition, which pozzolanic material comprises a mixture of metakaolin and from 2% to 50% by weight of an aqueous medium, based on the total weight of dry metakaolin and the aqueous medium.

The metakaolin is preferably prepared by calcining a kaolinitic clay at a temperature in the range of from 550°C to 925°C and for a time such that the loss on ignition for 2 hours at 1000°C is less than 1% by weight. Metakaolin prepared in this way is in the form of a substantially anhydrous powder, but it may contain up to about 1% by weight of water as a result of absorbing humidity from the air.

Preferably the metakaolin is mixed with from 5% to 40% by weight of the aqueous medium, based on the total weight of dry metakaolin and the aqueous medium. Such a mixture will still have the appearance of a dry powder. Most

preferably, the metakaolin is mixed with from 10% to 25% by weight of the aqueous medium, based on the total weight of dry metakaolin and the aqueous medium.

Advantageously the aqueous medium will be water. Alternatively the aqueous medium may be an aqueous solution containing as the solute, for example, an alkali, a dispersing agent, or an agent which either accelerates or retards the setting of the concrete. The addition of a solution of an alkali is found to be advantageous, because the rate of the pozzolanic reaction is increased if the pH of the aqueous medium is increased to within the range of from about 8 to about 12. The addition of a solution of a dispersing agent is advantageous because it improves the flow properties of the uncured concrete composition.

In the accompanying drawing, the single Figure is a graph showing the pozzolanic activity of a sample of metakaolin as a function of the moisture content of the metakaolin.

The invention will now be illustrated by reference to the following example:

EXAMPLE

Metakaolin was prepared by calcining a paper coating quality kaolin clay having a particle size distribution such that 80% by weight consisted of particles having an equivalent spherical diameter smaller than 2μm at a temperature of 500°C for 30 minutes. The loss on ignition at 1000°C for 2 hours of the metakaolin was 0.3% by weight. The metakaolin product was in the form of a substantially dry powder having a particle size distribution such that 50% by weight con- sisted of particles having an equivalent spherical diameter smaller than 2μm. The metakaolin was divided into a number of portions each of which was mixed with a different quantity of water to give mixtures of

metakaolin and water having moisture contents in the range from 0% by weight to 33% by weight, based on the total weight of metakaolin and water. The reactivity of each mixture with calcium hydroxide was determined by a method which is generally known in the field of concrete technology as the "Chappelle test". In this test a dilute slurry of the pozzolanic material is reacted with excess calcium hydroxide at 95°C for 18 hours. At the end of this period, the amount of unreacted calcium hydroxide is determined by titration. The procedure is described in detail by R.Largent, Bull. Liaison Lab. Ponts et Chaussées, Vol. 93 (1978), page 63. The results are shown on the accompanying Figure in which the graph shows the relationship between the weight of calcium hydroxide in milligrams absorbed by one gram of metakaolin and the percentage by weight of water in the metakaolin. It can be seen that the addition of from about 5% by weight to about 25% by weight of water to the metakaolin gives a very advantageous increase in the reactivity of the metakaolin with calcium hydroxide.

CLAIMS:

1. A pozzolanic material for incorporation in a concrete composition, which pozzolanic material comprises a mixture of metakaolin and from 2% to 50% by weight of an aqueous medium, based on the total weight of dry metakaolin and the aqueous medium.

2. A pozzolanic material according to claim 1, wherein the pozzolanic material comprises a mixture of metakaolin and from 5% to 40% by weight of an aqueous medium, based on the total weight of dry metakaolin and the aqueous medium.

3. A pozzolanic material according to claim 2, wherein the pozzolanic material comprises a mixture of metakaolin and from 10% to 25% by weight of an aqueous medium, based on the total weight of dry metakaolin and the aqueous medium.

4. A pozzolanic material according to claim 1, 2 or 3, wherein the aqueous medium is water.

5. A pozzolanic material according to claim 1, 2 or 3, wherein the aqueous medium is an aqueous solution.

6. A pozzolanic material according to claim 5, wherein the aqueous solution contains as the solute an alkali, a dispersing agent or an agent which either accelerates or retards the setting of the concrete.

7. A pozzolanic material according to any one of claims 1 to 6, wherein the metakaolin is prepared by calcining a kaolinitic clay at a temperature in the range of from 550°C to 925°C and for a time such that the loss on ignition for 2 hours at 1000°C is less than 1% by weight.

The Patent Office

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Application No: GB 9421046.5
Claims searched: 1 to 7

Examiner: Miss M. M. Kelman
Date of search: 7 July 1995

Patents Act 1977
Search Report under Section 17

Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

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Other: ONLINE:PATENTS

Documents considered to be relevant:

Category	Identity of document and relevant passage		Relevant to claims
X	US 5282898 A	ENGELHARD CORPORATION see claims 1 to 5 and column 5, line 47 to column 6, line 31	1,2,4,6,7
X	US 5261956 A	ECC INTERNATIONAL see claim 8 and column 1, line 63 to column 2, line 4	1,2,4,7
X	US 5232881 A	ENGELHARD CORPORATION see claims 1 to 6 and 15 to 18 and column 6, lines 6 to 22	1,2,4,5,6,7
X	US 5129553 A	E.C.C. AMERICA see column 1, line 63 to column 2, line 9 and column 4, lines 56 to 60	1,4,7
X	US 5074475 A	E.C.C. AMERICA see claims 1 to 7, column 1, line 61 to column 2, line 2 and column 5, lines 23 to 31	1,4,5,6,7
X	US 5028268 A	E.C.C. AMERICA see the claims, column 1, lines 59 to 68 and column 5, lines 23 to 30	1,4,5,6,7

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
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